Docket No.: 28682/38519
PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Utility Application of: Kanzunobu Maruo, et al.	
Application No.: 10/743,115	Confirmation No.: 8485
Filed: December 22, 2003	Group Art Unit: 1796
Attorney Docket No.: 28682/38519	Examiner: V. M. Ronesi
For: OLIGOMER-MODIFIED LAYERED INORGANIC COMPOUNDS AND THEIR USE IN NANOCOMPOSITES	

Commissioner for Patents
Washington, DC 20231

Dear Madam:

DECLARATION OF TIE LAN UNDER 37 CFR § 1.132

I, Tie Lan, one of the named inventors of the subject matter of this patent application and the General Manager of Nanocor, Inc., a subsidiary of the Assignee of the above-identified patent application, do hereby declare and state the following:

1. My education since high school is as follows:

BS Chemistry - 1986	Beijing University, Beijing, China
MS Chemistry - 1989	Beijing University, Beijing, China
Ph.D. Chemistry - 1995	Michigan State University, East Lansing, MI "Polymer-Clay Nanocomposites"

2. My work experience is as follows:

1995-1996	Research Associate	Michigan State University
1996-1998	Research Scientist	Nanocor, Inc.
1999-2001	R&D Manager	Nanocor, Inc.
2001-2006	Technical Director	Nanocor, Inc

2006-Present General Manager Nanocor, Inc.

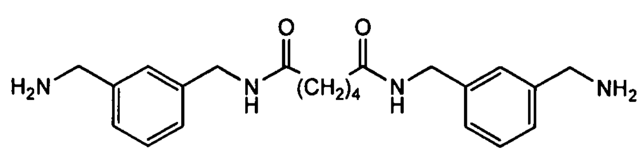
3. I have published more than 40 papers and patents on polymer-clay nanocomposites.

4. I have studied the Office Actions dated February 10, 2009 and January 17, 2007 and the invention disclosed and claimed in Barbee et. al., U.S. Pat. No. 6,384,121 ("Barbee").

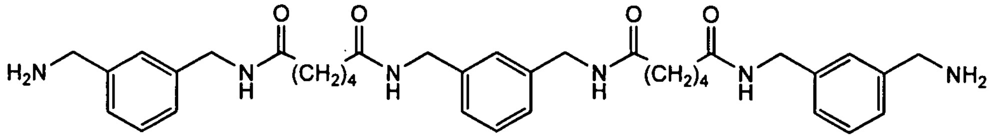
5. As set forth in the Office Action dated January 17, 2007, the prior art Barbee reference discloses in example 24 an exfoliated nanocomposite having a octadecylammonium intercalated montmorillonite clay, a trimethylammonium functionalized poly(*m*-xylene adipamide) and a MXD6 polymer.

6. In laboratories that I manage, I had manufactured and tested for me: BAB intercalated, BABAB intercalated, and surfactant intercalated clay materials. BAB and BABAB correspond to the materials recited in the above referenced patent application and shown in Figure 1, below. The intercalated clays were intercalated montmorillonite prepared under procedures known in the art or recited in the above referenced patent application. Among the materials tested was a sample of the octadecyl ammonium intercalated montmorillonite clay disclosed in the Barbee reference. *See* Barbee, Col. 19, lines 46-47. A list of the materials manufactured and tested for me appear in Table 1.

FIGURE 1.



BAB



BABAB

TABLE 1.

	Sample	Results
1	Clay intercalated with ***octadecyl trimethyl ammonium***	Exhibit 1
2	Clay intercalated with ***octadecyl dimethyl benzyl ammonium***	Exhibit 2
3	Clay intercalated with ***dehydrogenated tallow dimethyl ammonium***	Exhibit 3
4	Clay intercalated with ***octadecyl ammonium***	Exhibit 4
5	Clay intercalated with ***BAB***	Exhibit 5
6	Clay intercalated with ***BABAB***	Exhibit 6

7. I had the thermal stability of the six prepared samples tested by thermal gravimetric analysis ("TGA"). This procedure measures the change in the mass of a sample as the temperature of the sample and surrounding environment is increased. The technique is a standard method for determining the thermal stability of a material, showing in addition to the loss of volatile components, e.g. water at 100 °C, the pyrolysis and/or combustion of the material, here I will refer to both processes as pyrolysis. Test results are attached as Exhibits 1-6.

8. In comparison, the pyrolysis of the BAB and BABAB intercalated clays occurred at a surprisingly high temperature; this result was unexpected and is very important industrially. As can be see in Exhibits 1-4 the pyrolysis of clays intercalated with ammonium based surfactants showed an onset of pyrolysis below 300 °C. For example, the octadecyl ammonium intercalated montmorillonite used in the Barbee patent (at Col. 19, lines 46-47) showed an onset of pyrolysis at 248 °C and a significant weight loss corresponding to the decomposition of the intercalant (here the octadecyl ammonium). The pyrolysis onset temperatures for the intercalated clays samples were obtained by TGA and are listed in Table 2.

TABLE 2.

	Sample	Pyrolysis Onset Temperature (°C)
1	Clay intercalated with ***octadecyl trimethyl ammonium***	238
2	Clay intercalated with ***octadecyl dimethyl benzyl ammonium***	220
3	Clay intercalated with ***dehydrogenated tallow dimethyl ammonium***	248
4	Clay intercalated with ***octadecyl ammonium***	248
5	Clay intercalated with ***BAB***	318
6	Clay intercalated with ***BABAB***	310

9. Commonly, polyamides are melt processed at high temperatures, generally, at least 15 to 60 °C above their melting points. For example, Nylon 6 melts at about 215 °C and Nylon 6,6 melts at about 260 °C and these polyamides are processed at about 230-275 and 275-320 °C, respectively. By comparison, the Barbee patent processed the MXD6 polyamide at 280 °C.

10. High polyamide processing temperatures, e.g. 280 °C, can lead to the pyrolysis of materials intercalated into clays, as shown by the TGA data above. The pyrolysis of the intercalated material creates significant unintended consequences in any final product. Some problems created by the pyrolysis of an intercalant during the melt processing of a polyamide include the discoloration of the material, the formation of an odor, an color and the formation of chemical compounds with unknown toxicities.

11. The unexpectedly high thermal stability of samples 5 and 6 make these samples industrially desirable. One example of the industrial applicability of these samples is

in materials that contact foods. Nylon resins are approved substances for use as basic components of single and repeated use food contact surfaces. See 21 CFR 177.1500 (2008). The FDA requirements limit the additives permitted in the nylon to, in part, those "substances generally recognized as safe for use in food and food packaging." 21 CFR 177.1500(c)(3)(iv)(A). While clays, like montmorillonite, are recognized as safe for use in food and food packaging, the pyrolysis products of intercalated surfactants are not. Therefore, the food packaging applicability of intercalated clays as a nylon additive requires the nylon additive's thermal stability to exceed the nylon processing temperature. As shown, only the BAB and BABAB intercalated clays satisfy this requirement and are applicable in the food industry.

14. All statements made herein of my own knowledge are true, and that all statements made upon information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like, so made are punishable by fine or imprisonment, or both, under section 1001 of title 10 of the United States Code and that such willful false statements may jeopardize the validity of the instant patent application or any patent issuing thereon.

Lan Tie

March 10, 2009
Date

Tie Lan

EXHIBIT 1

In re Utility Application of Kanzunobu Maruo, et al.
Application No.: 10/743,115
Filed: December 22, 2003

TGA DATA FOR A MONTMORILLONITE CLAY INTERCALATED WITH *OCTADECYL TRIMETHYL AMMONIUM.*

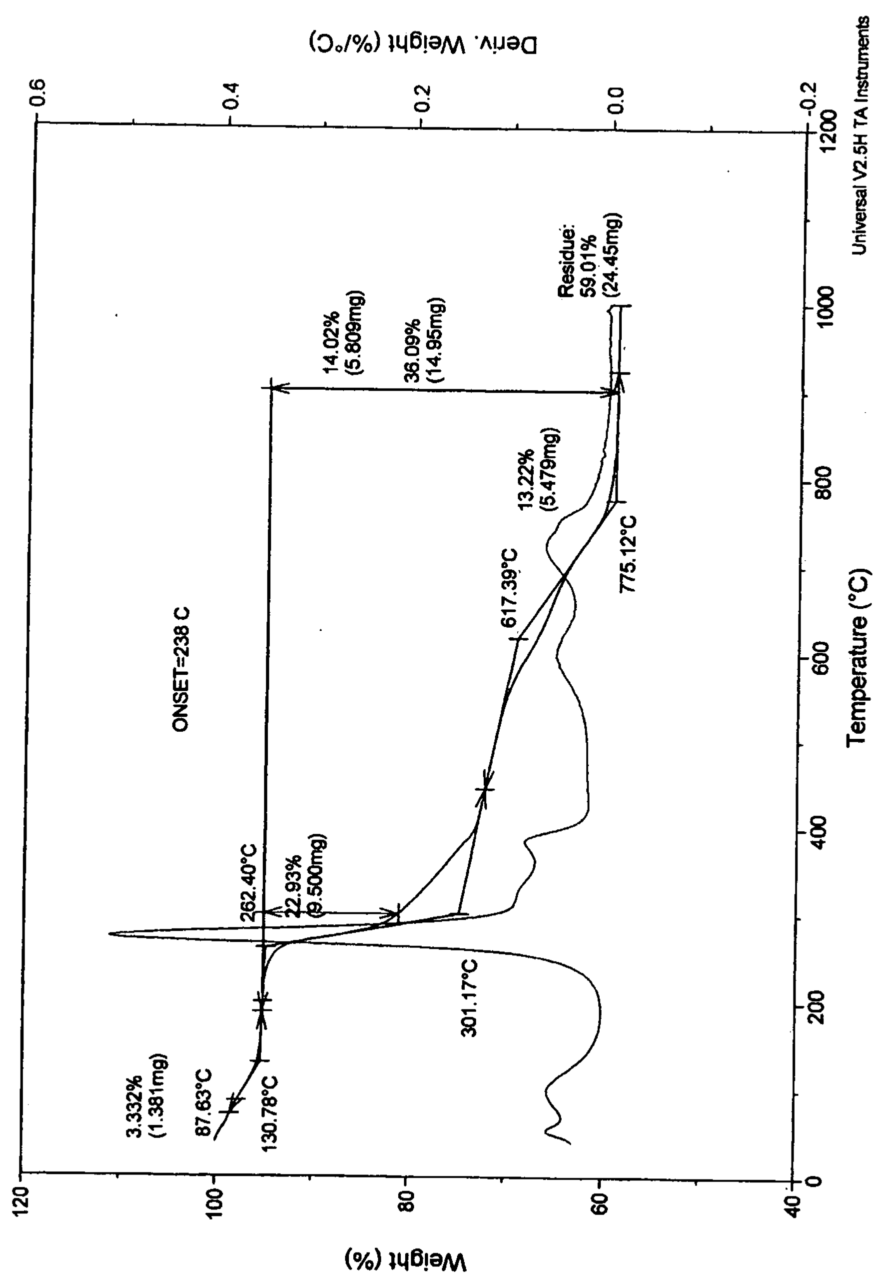


EXHIBIT 2

In re Utility Application of Kanzunobu Maruo, et al.
Application No.: 10/743,115
Filed: December 22, 2003

TGA DATA FOR A MONTMORILLONITE CLAY INTERCALATED WITH *OCTADECYL DIMETHYL BENZYL AMMONIUM.*

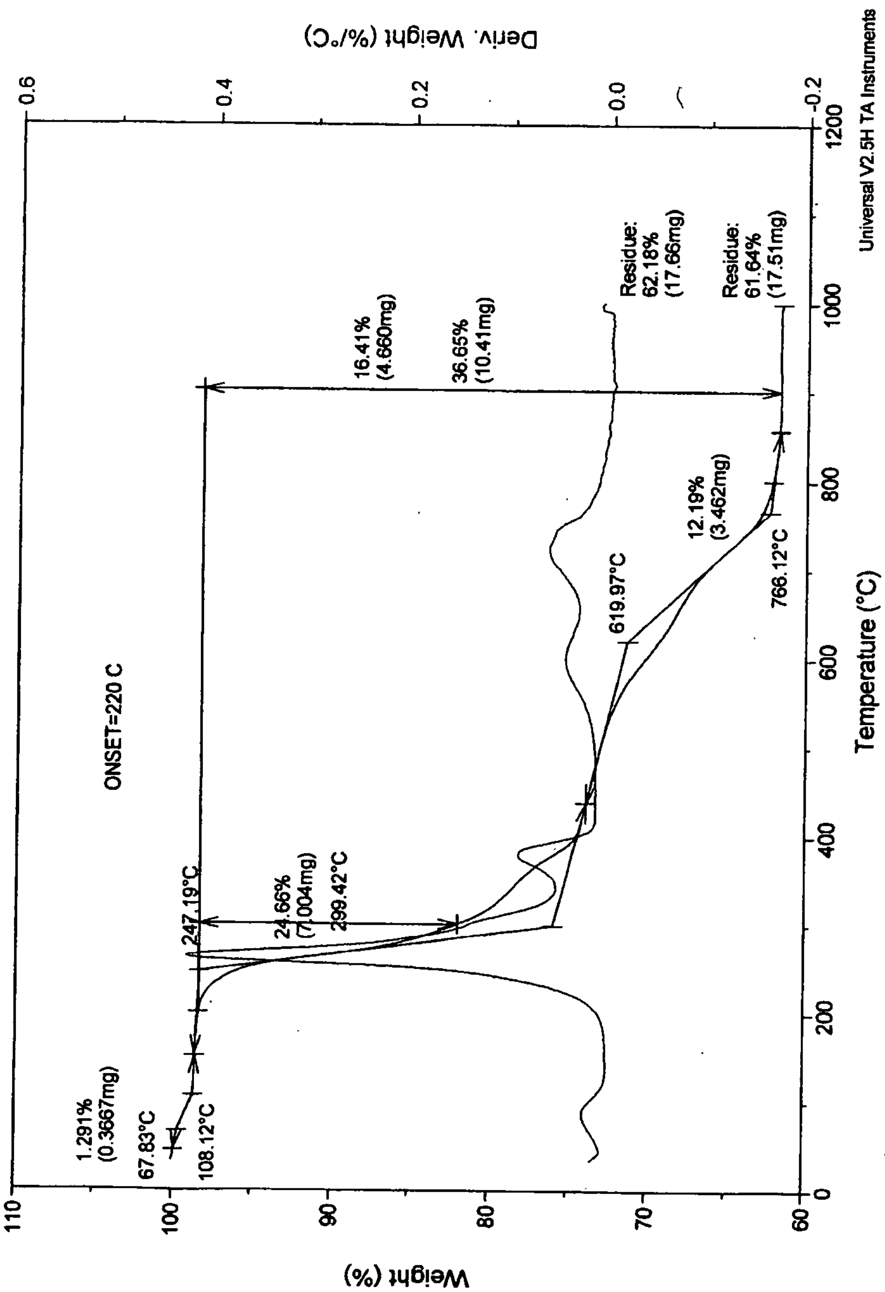


EXHIBIT 3

In re Utility Application of Kanzunobu Maruo, et al.
Application No.: 10/743,115
Filed: December 22, 2003

TGA DATA FOR A MONTMORILLONITE CLAY INTERCALATED WITH
DEHYDROGENATED TALLOW DIMETHYL AMMONIUM.

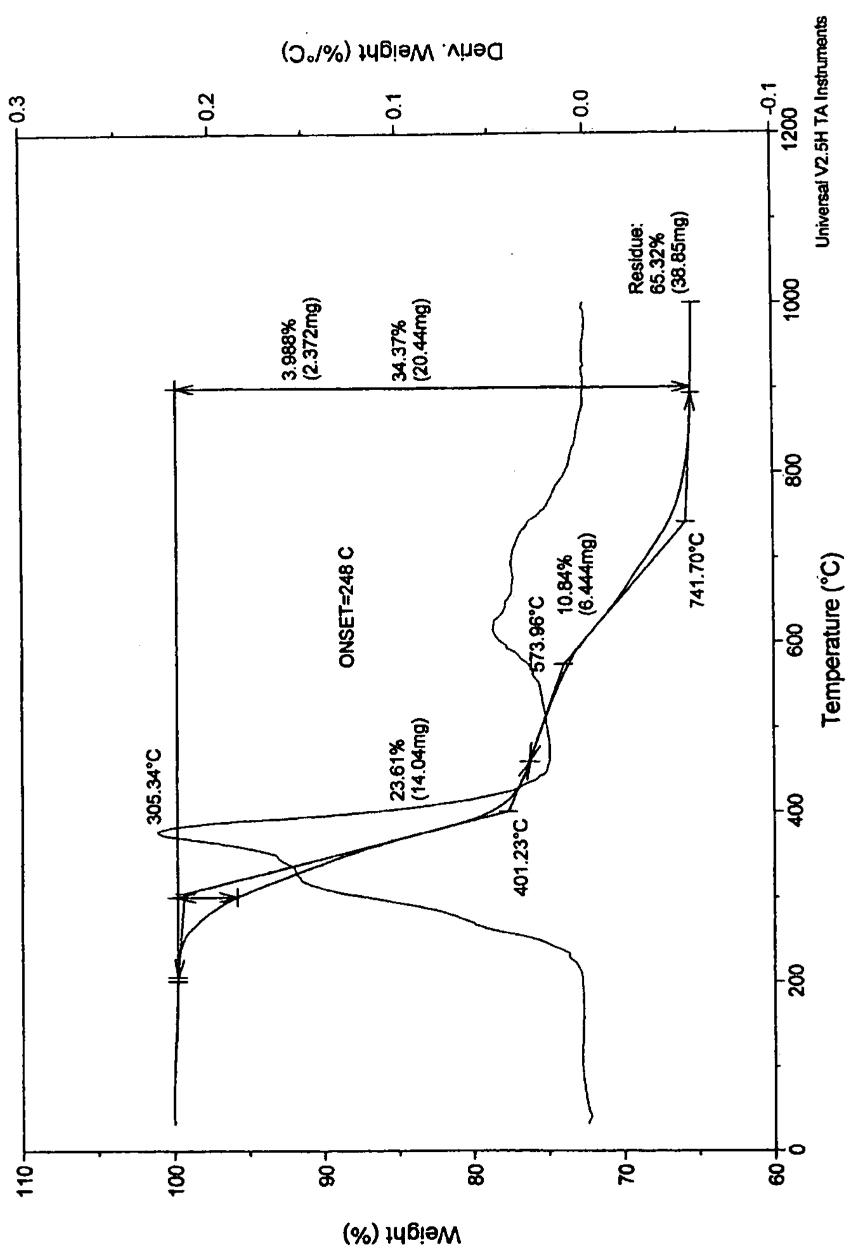


EXHIBIT 4

In re Utility Application of Kanzunobu Maruo, et al.
Application No.: 10/743,115
Filed: December 22, 2003

TGA DATA FOR A MONTMORILLONITE CLAY INTERCALATED WITH *OCTADECYL AMMONIUM.*

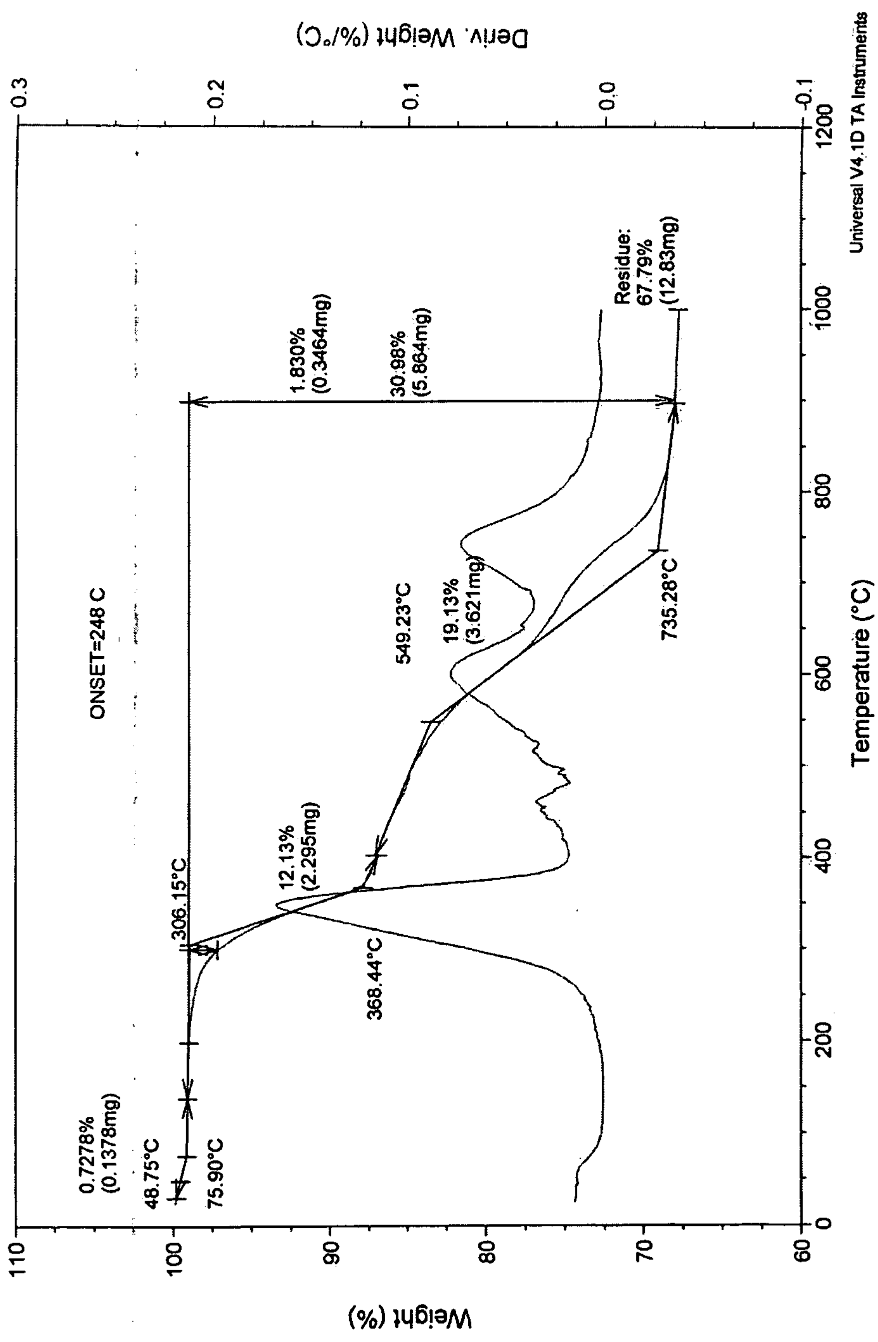


EXHIBIT 5

In re Utility Application of Kanzunobu Maruo, et al.
Application No.: 10/743,115
Filed: December 22, 2003

TGA DATA FOR A MONTMORILLONITE CLAY INTERCALATED WITH *BAB.*

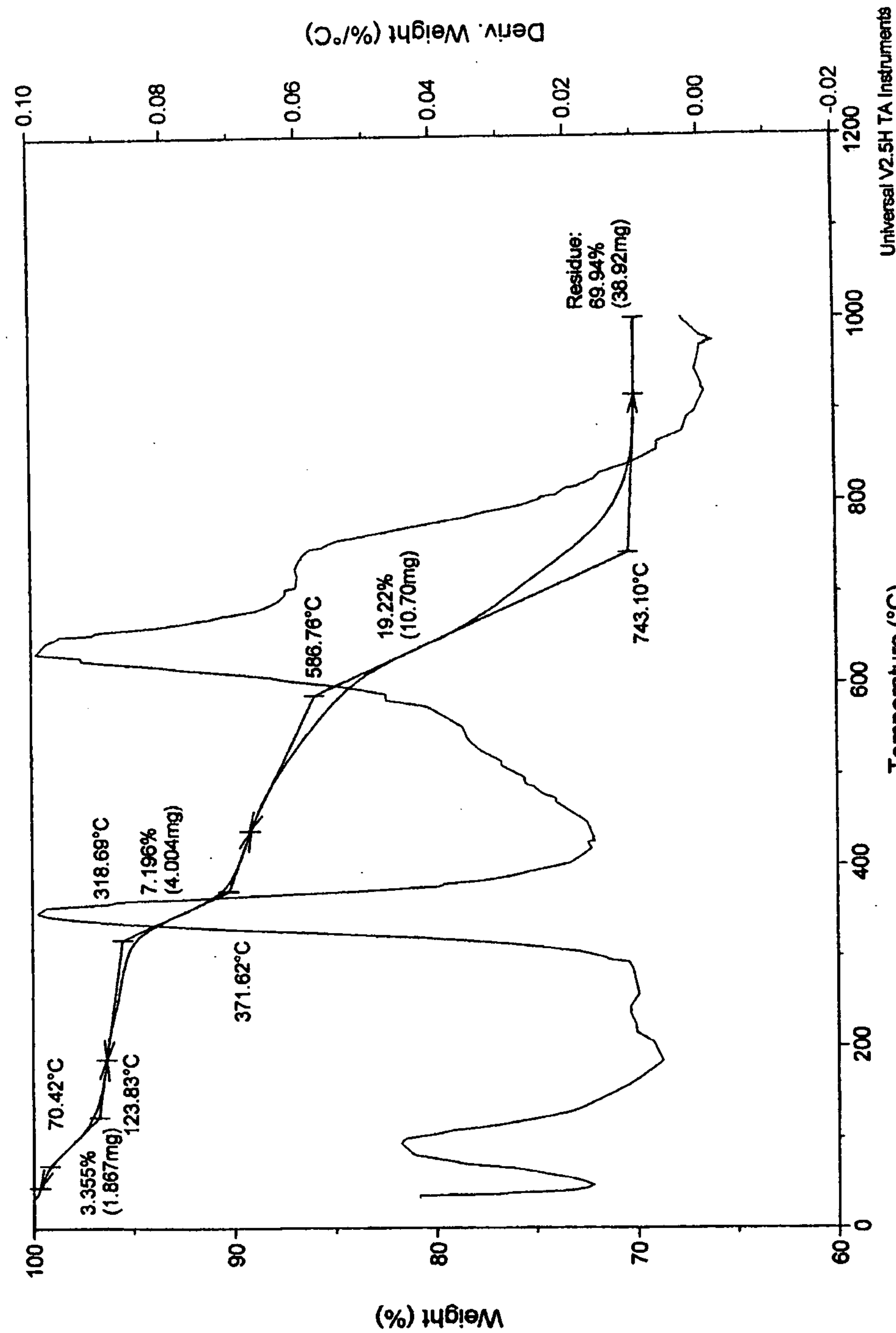


EXHIBIT 6

In re Utility Application of Kanzunobu Maruo, et al.
Application No.: 10/743,115
Filed: December 22, 2003

TGA DATA FOR A MONTMORILLONITE CLAY INTERCALATED WITH *BABAB.*